

LA-UR-17-27976

Approved for public release; distribution is unlimited.

Title: 3D Microstructural Characterization of Uranium Oxide as a Surrogate
Nuclear Fuel: Effect of Oxygen Stoichiometry on Grain Boundary
Distributions

Author(s): Rudman, K
McClellan, Kenneth James
Dickerson, P
Byler, Darrin David
Peralta, P
Lim, H
McDonald, R
Dickerson, R

Intended for: unknown conference proceedings

Issued: 2017-09-06

Disclaimer:

Los Alamos National Laboratory, an affirmative action/equal opportunity employer, is operated by the Los Alamos National Security, LLC for the National Nuclear Security Administration of the U.S. Department of Energy under contract DE-AC52-06NA25396. By approving this article, the publisher recognizes that the U.S. Government retains nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or to allow others to do so, for U.S. Government purposes. Los Alamos National Laboratory requests that the publisher identify this article as work performed under the auspices of the U.S. Department of Energy. Los Alamos National Laboratory strongly supports academic freedom and a researcher's right to publish; as an institution, however, the Laboratory does not endorse the viewpoint of a publication or guarantee its technical correctness.

3D Microstructural Characterization of Uranium Oxide as a Surrogate Nuclear Fuel: Effect of Oxygen Stoichiometry on Grain Boundary Distributions

K. Rudman¹, P. Dickerson², D. Byler², P. Peralta¹, H. Lim¹, R. McDonald¹, R. Dickerson², K. McClellan²

¹Arizona State University, Tempe, Arizona 85287

²Los Alamos National Laboratory, Los Alamos, New Mexico 87545

The initial microstructure of an oxide fuel can play a key role in its performance. At low burn-ups, the diffusion of fission products can depend strongly on grain size and grain boundary (GB) characteristics, which in turn depend on processing conditions and oxygen stoichiometry. Serial sectioning techniques using Focused Ion Beam were developed to obtain Electron Backscatter Diffraction (EBSD) data for depleted UO_2 pellets that were processed to obtain 3 different oxygen stoichiometries. The EBSD data were used to create 3D microstructure reconstructions and to gather statistical information on the grain and GB crystallography, with emphasis on identifying the character (twist, tilt, mixed) for GBs that meet the Coincident Site Lattice (CSL) criterion as well as GBs with the most common misorientation angles. Data on dihedral angles at triple points were also collected. The results were compared across different samples to understand effects of oxygen content on microstructure evolution.

The 3D reconstruction of the microstructure of an initial sample of d- $\text{UO}_{2.1}$ has been done and the model currently consists of 87 Focused Ion Beam (FIB) slices separated by 0.5 μm , with a total of over 300 grains. This provides enough grains and grain boundaries (GBs) to obtain reliable statistics of correlations between microstructural features, local crystallography and local porosity. The 3-D reconstruction shows that the location of the pores plays an important role in the grain growth and shape. Some large rounded pores located at the grain boundaries (GBs) pin them seem to restrict the local grain growth, which affects the grain shape and leads to local convexity in the GB curvature. This phenomenon was observed in several grains in the microstructure and further studies will benefit from higher resolution FIB serial sectioning in smaller volumes, so the pores can be resolved in 3-D, which is not the case with the existing model.

References:

- [1] Nerikar, P.V. ; Rudman, K. ; Desai, T.G. ; Byler, D. ; Unal, C. ; et al. in Journal of the American Ceramic Society (2011) Vol. 94, iss. 6, p.1893-1900
- [2] Acknowledgments: Funded as part of the Advanced Fuels Development program at Los Alamos National Laboratory

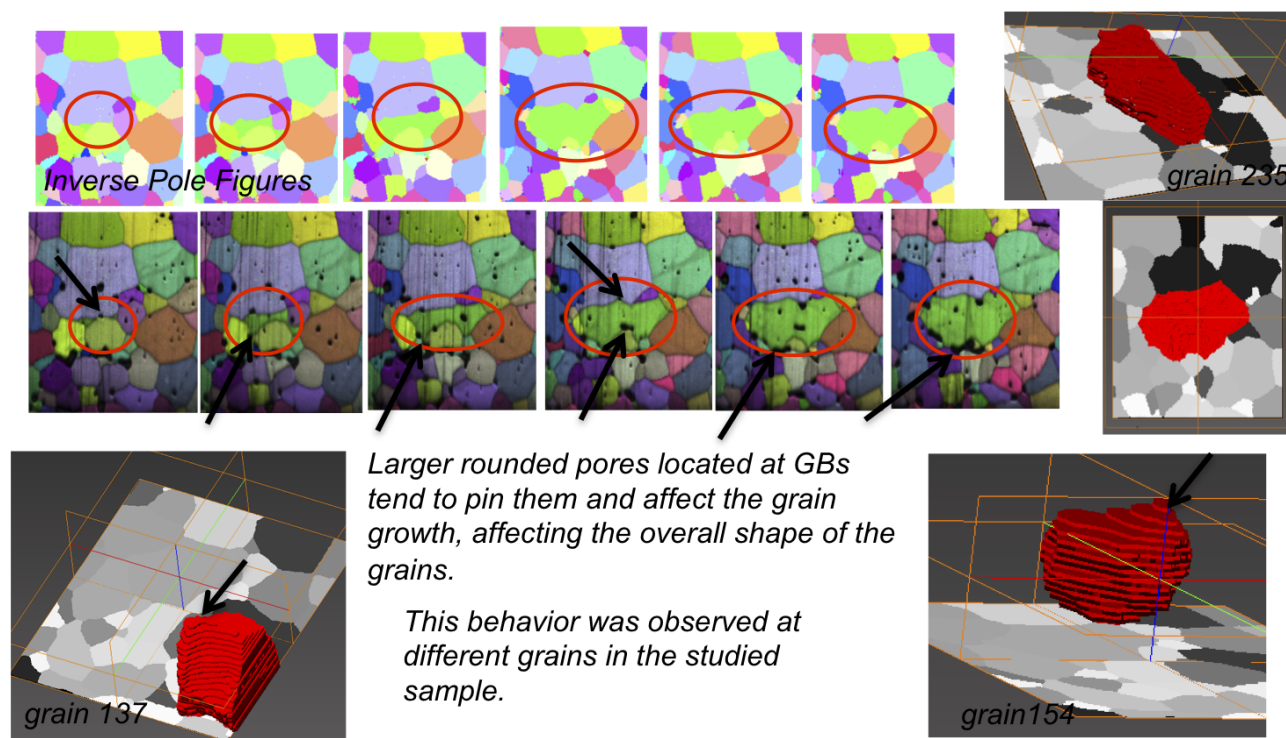
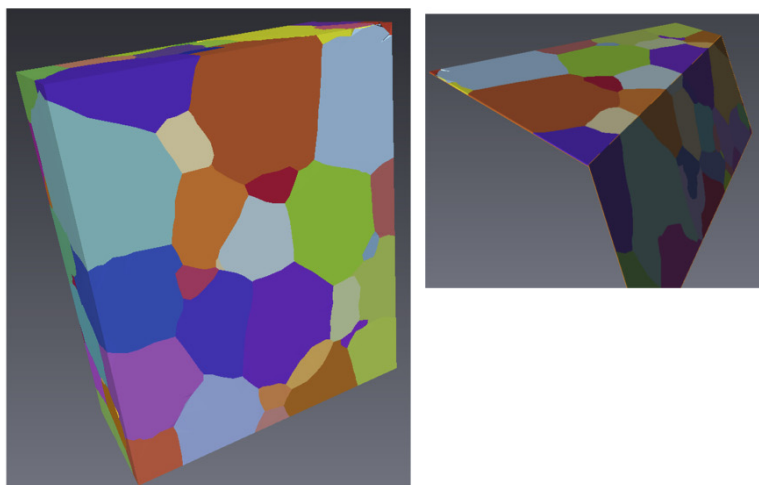
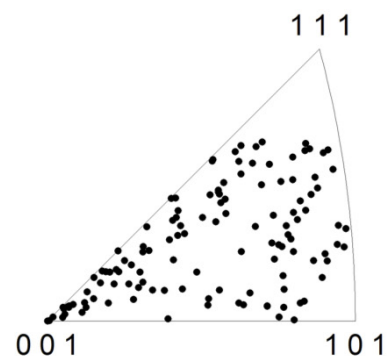


Fig. 1. Inverse pole figure and image quality maps of the microstructure of a sample of $d\text{-UO}_{2+x}$ ($x \approx 0.1$).

The 3D reconstructed microstructure was also used to determine the GB normals in order to fully characterize the 5 degrees of freedom of a bicrystalline interface



[001]



Grain boundary normals with respect to crystallographic coordinates plotted in an IPF. Note the absence of normals close to $\{111\}$ and $\{110\}$

Fig. 2. AVIZO™ reconstructed surface and its cutting planes that reveal grain boundary normals.

Reproduced with permission of the copyright owner. Further reproduction prohibited without permission.